

**A HOLDER FOR USE IN SEMICONDUCTOR DEVICE  
MANUFACTURING AND BIO-MEDICAL SAMPLE PROCESSING**

**CROSS-REFERENCES TO RELATED APPLICATIONS**

[0001] This is a continuation-in-part of Application No. 09/658,185 filed September 8, 2000, the disclosure of which is incorporated herein by reference. Application No. 09/658,185 was filed under 35 U.S.C. § 111(a) claiming benefit pursuant to 35 U.S.C. § 119(e)(1) of the filing date of Provisional Application No. 60/206,726 filed on May 25, 2000 pursuant to 35 U.S.C. § 111(b).

**BACKGROUND OF THE PRESENT INVENTION**

1. Technical Field of the Present invention

[0002] This present invention relates to improvements in holders for sample preparation for electron microscopy and semiconductor wafer manufacturing. . The present invention is embodied in a holder that holds semiconductor wafers in drying apparatuses and in liquid baths during the fabrication process of the wafers leading up to critical point drying, and alternatively provides fluid flow around the wafers or a containment system to keep the wafer submerged in fluid.

2. Description of the Related Art

[0003] The following references provide useful background information on the indicated topics, all of which relate to the present invention, and are incorporated herein by reference:

U.S. Patent No. 4,055,904 issued to Horne on November 1, 1977 describes an automatic method of operating the purge and bleed modes for a critical point dryer.

U.S. Patent No. 4,104,808 issued to Horne et al. on August 8, 1978 describes a critical point dryer wherein the purge and bleed modes are controlled semi-automatically.

[0004] There will now be provided a discussion of various topics to provide a proper foundation for understanding the present invention.

[0005] In order to examine biological specimens under a scanning electron microscope, the biological specimens must be completely dried and coated with a thin conductive layer. It is important that the drying process be accomplished without disturbing the microstructure of the biological specimen to be examined. Depending upon the biological specimen's structure, three techniques are available for drying the biological specimen. The first method is air drying by evaporation of the cellular water. While suitable for bacteria or other rigid structures, this method is detrimental to the structures of many biological specimens. The surface tension forces, which turn a grape into a raisin during the drying process, cause sufficient distortion in the cell structure of many biological specimens thereby rendering them useless. The second method is sublimation or freeze-drying. This method is useful only for very small specimens. Additionally, unless the lengthy technique is followed precisely, structural damage from thermal expansion or ice crystal formation often results. The third method utilized is the phase transitional or critical point drying which produces consistently reproducible results without the drawbacks of the preceding two methods.

[0006] Along with being used to prepare specimens for the scanning electron microscope, critical point drying may also be used in the production of MEMS (Micro- Electro-Mechanical Systems) devices. The critical point drying process helps for a sticktion free release of microstructers in the MEMS device.

[0007] In critical point drying, a dehydrating fluid such as ethanol or acetone gradually replaces the water contained in a specimen. This maintains the three-dimensional hydrated morphology of the structure under study. However, if the ethanol or acetone evaporates, surface tension forces would cause structural damage and destroy the specimen's usefulness.

[0008] Critical point drying devices for sample preparation in electron microscopy are known in the art. The prior art critical point dryers utilize the technique of substituting a transitional fluid for the dehydrating fluid in the cell structure and then removing the transitional fluid. A critical point dryer heats and pressurizes the biological specimen until above the critical pressure and critical temperature. The critical temperature is defined as the temperature above which a gas cannot be liquefied by pressure alone. The critical pressure is the pressure that results when a substance exists as a gas and a liquid in equilibrium at the critical temperature. The critical point of a liquid is when its temperature and pressure are at or above the critical temperature and pressure and the densities of the liquid phase and vapor phase are identical. This critical point is characterized by an absence of phase boundaries that normally exist between a liquid and its vapor at temperatures and pressures below the critical point. This absence of a phase boundary eliminates the boundary forces that exist when changing a liquid

to a gas. These boundary forces often cause the destruction of the extremely delicate specimens when changing its internal liquid to a gas below the critical point. Therefore, the solution which is applied in a critical point drying process is to remove the internal liquid from the biological specimen above its critical pressure and temperature to eliminate the boundary force destruction that would otherwise result.

[0009] Although all fluids have a characteristic critical point which should allow direct removal without the use of dehydrating or transitional fluids, the critical point temperature and pressure of water is 374.2° C and 218 atmospheres. Achieving these temperatures and pressures would cause severe damage to most biological specimens and therefore a fluid having a lower critical temperature and pressure is normally substituted. Commonly, a dehydrating fluid is used that is miscible with water (e.g., ethanol or acetone) as an intermediate stage between the specimen containing water and a specimen containing transitional fluid.

[0010] Typically, and in the prior art dryers, the transitional fluid commonly used is carbon dioxide (CO<sub>2</sub>) because it is easy to use, more economical, less noxious and provides consistently better results than other transitional fluids. The critical temperature and pressure of carbon dioxide is 31° C and 1,072 psi, respectively, thus reducing the potential for destruction of the specimen structure.

[0011] The known instruments and apparatuses for critical point drying of biological specimens include, of course, a drying chamber that is connected a supply of the transitional fluid with various regulating valves, temperature gauges

and a means for heating the chamber. A skilled technician must carefully control the application, heating, pressurizing and removal of the transitional fluid, thus requiring not only time but also constant attention.

#### SUMMARY OF THE PRESENT INVENTION

[0012] The present invention has been made in view of the above circumstances and has an object to overcome the above problems and limitations of the prior art.

[0013] Additional objects and advantages of the present invention will be set forth in part in the description that follows and in part will be obvious from the description, or may be learned by practice of the present invention. The objects and advantages of the present invention may be realized and attained by means of the instrumentalities and combinations particularly pointed out in the appended claims.

[0014] According to a first aspect of the invention, a critical point drying chamber for drying specimens comprising a chamber and at least one heater is provided. The apparatus further comprises a first valve assembly that supplies a cooling fluid to the chamber. The apparatus further comprises a second valve assembly that supplies a transitional fluid having a critical point temperature and critical point pressure to the chamber. The apparatus further comprises a third valve assembly that allows an intermediary fluid to be purged from the chamber. The apparatus further comprises a fourth valve assembly that bleeds the transitional fluid from the chamber. The apparatus further comprises a condenser collector that collects intermediary fluid exiting the third valve assembly.

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[0015] According to a second aspect of the invention, a critical point drying apparatus for drying specimens comprises a cylindrical drying chamber having concave end portions and at least one heater. The apparatus further comprises a first valve assembly that supplies a cooling fluid to the drying chamber. The apparatus further comprises a second valve assembly that supplies a transitional fluid having a critical point temperature and critical point pressure to the drying chamber. The apparatus further comprises a third valve assembly that allows an intermediary fluid to be purged from the drying chamber. The apparatus further comprises a fourth valve assembly that bleeds the transitional fluid from the drying chamber.

[0016] According to a third aspect of the invention, a critical point drying apparatus for drying specimens comprises a drying chamber having at least one heater. The apparatus further comprises a first valve assembly that supplies a cooling fluid to the drying chamber. The apparatus further comprises a second valve assembly that supplies a transitional fluid having a critical point temperature and critical point pressure to the drying chamber. The apparatus further comprises a third valve assembly that allows an intermediary fluid to be purged from the drying chamber. The apparatus further comprises a fourth valve assembly that bleeds the transitional fluid from the drying chamber. The apparatus further comprises a computer system that operates the first, second, third and fourth valve assemblies and that activates the at least one heater to heat the transitional fluid above the critical point temperature and to pressurize the transitional fluid above the critical point pressure.



critical point temperature and critical point pressure while maintaining the drying chamber at the first chamber temperature such that the transitional fluid completely displaces an intermediary fluid within a first time period. The software instructions further enable the computer system to activate at least one heater to raise the transitional fluid to its critical point pressure and critical point temperature, thereby reaching critical point equilibrium. The software instructions further enable the computer system to maintain the transitional fluid at the critical point equilibrium for a second time period. The software instructions further enable the computer system to bleeding the transitional fluid from the drying chamber while maintaining the drying chamber at the second chamber temperature and allowing the drying chamber pressure to drop.

[0019] According to a sixth aspect of the invention, a computer program product for enabling a computer system to control the drying chamber during a critical point drying process is provided. The computer program product comprises software instructions for enabling the computer system to perform predetermined operations, and a computer readable medium bearing the software instructions. The predetermined operations comprise cooling the drying chamber to a first chamber temperature. The predetermined operations comprise filling the drying chamber with a transitional fluid having a critical point temperature and critical point pressure while maintaining the drying chamber at the first chamber temperature such that the transitional fluid completely displaces the intermediary fluid within a first time period. The predetermined operations comprise activating at least one heater to raise the transitional fluid to its critical

point pressure and critical point temperature, thereby reaching critical point equilibrium. The predetermined operations comprise maintaining the transitional fluid at the critical point equilibrium for a second time period. The predetermined operations comprise bleeding the transitional fluid from the drying chamber while maintaining the drying chamber at the second chamber temperature.

[0020] According to a seventh aspect of the invention, an article of manufacture, which comprises a computer readable medium having stored therein a computer program to control a drying chamber during a critical point drying process, is provided. The article of manufacture comprises a first code segment which, when executed on a computer, cools the drying chamber to a first chamber temperature. The article of manufacture comprises a second code segment which, when executed on a computer, fills the drying chamber with a transitional fluid having a critical point temperature and critical point pressure while maintaining the drying chamber at the first chamber temperature such that the transitional fluid completely displaces the intermediary fluid within a first time period. The article of manufacture comprises a third code segment which, when executed on a computer, activates at least one heater to raise the transitional fluid to its critical point pressure and critical point temperature, thereby reaching a critical point equilibrium. The article of manufacture comprises a fourth code segment which, when executed on a computer, maintains the transitional fluid at the critical point equilibrium for a second time period. The article of manufacture comprises a fifth code segment which, when executed on a computer, bleeds the transitional

fluid from the drying chamber while maintaining the drying chamber at the second chamber temperature.

[0021] According to an eighth aspect of the invention, a holder may be provided for use in the critical point drying chamber, although it is not limited to such an application, and is suitable for use in, for example only, other drying chambers, or other types of fluid baths. The holder comprises an outer section and a generally cup shaped inner base section. The outer section comprises a handle and a closing wall, while the inner base section comprises a retention wall, flow holes and/or standoffs.

[0022] The holder has two positions corresponding to a position to allow fluid flow, and a position to provide fluid containment. When the holder is in a position to provide fluid containment, the outer section is placed over the inner base section, which allows the closing wall to close off the flow holes in such a way as to prevent any fluid contained within the inner base section to escape. The closing wall has only a slightly larger diameter than the retention wall, in order to provide a tight fit and allow the holder to be moved by an operator using the handle.

[0023] When the holder is in a position to allow fluid flow, the outer section is removed from interaction with inner base section, causing the flow holes to not be covered by the annular retention wall, and allowing fluid to flow in and out of the holes.

[0024] According to a ninth aspect of the invention, the holder comprises a holding section, which may include a handle. The holding section comprises

a generally cup shaped inner holding section with at least one flow hole, and an outer retaining section with at least one flow hole.

**[0025]** The holder has two positions corresponding to a position to allow fluid flow, and a position to allow fluid containment. When the holder is in a position to provide fluid containment, the outer retaining section is rotated around the inner holding section in such a way as to not allow the flow holes of each section to communicate with each other, and thus prevent fluid that is contained within the inner holding section to flow through the holes.

**[0026]** When the holder is in a position to allow fluid flow, the outer retaining section is rotated around the inner holding section in such a way as to allow the flow holes of each section to communicate with each other, thus allowing fluid to flow into and out of the fluid holes.

**[0027]** According to a tenth aspect of the invention, the holder comprises a generally cup shaped holding section, which may include a handle. The holding section comprises a bottom section with flow holes, a plug seal and a resilient member.

**[0028]** The holder has two positions corresponding to a position to allow fluid flow, and a position to provide fluid containment. When the holder is in a position to allow fluid containment, pressure is not applied to the holder, which allows the plug seal to remain seated in the fluid hole. When fluid flow is desired, pressure may be applied to holder, which causes the plug seal to unseal the flow hole and allow fluid to flow. The resilient member will return the plug seal to a position that seals the flow hole when pressure on the holder is removed.

[0029] According to an eleventh aspect of the invention, a holder comprises a generally cup shaped holding section and may include a handle. The holding section comprises a bottom section, at least one plug seal and at least one plug actuating surface. The bottom section has flow holes to provide fluid flow.

[0030] The holder has two positions corresponding to a position to allow fluid flow, and a position to provide fluid containment. When the holder is in a position to provide fluid containment, pressure is not applied to the plug actuating surface, which allows the plug seal to remain seated in the flow hole. When fluid flow is desired, pressure may be applied to the plug actuating surface by placing the holder into a position such that it is resting on an opposing surface which engages the plug actuating surface and causes the plug seal to move vertically upward, thereby unsealing the flow hole and allowing fluid to flow. The plug seal will return to a position that seals the flow hole when holder is removed from the opposing surface.

[0031] The above and other objects and advantages of the present invention will become apparent from the following detailed description and with reference to the accompanying drawing figures.

#### BRIEF DESCRIPTION OF THE DRAWINGS

[0032] The accompanying drawings, which are incorporated in and constitute a part of this specification illustrate embodiments of the present invention and, together with the written description, serve to explain the objects, advantages and principles of the present invention. In the drawings,

FIG. 1 illustrates a perspective view of a first embodiment of the invention;

FIG. 2 illustrates a perspective view of a second embodiment of the invention;

FIG. 3 is a top view of the drying chamber illustrating the location of the purge outlets, the fill inlets, the cool inlet and the cool outlet;

FIG. 4 is a top view of the chamber cover;

FIG. 5 illustrates a cross-sectional view along lines IV-IV of the chamber cover, the cover viewing port and the cover viewing window;

FIG. 6 illustrates a cross-sectional view of the drying chamber along lines I-I showing the fill inlets, the purge outlets, the chamber viewing port and the chamber viewing window;

FIG. 7 illustrates a cross-sectional view of the drying chamber along lines II-II showing the temperature sensor, the heat sensor, the cool sensor and the heater;

FIG. 8 illustrates a cross-sectional view of the drying chamber along lines III-III showing the cool inlet and the cool outlet;

FIG. 9 illustrates the various valves and connection lines for the routing of cooling fluid and transitional fluid through the critical point drying apparatus;

FIG. 10 illustrates the various valves and connection lines for the routing of cooling fluid and transitional fluid through a critical point drying apparatus that is connected to a closed loop refrigeration system;

FIG. 11 illustrates a side view of the collector condenser;

FIG. 12 illustrates the data flow paths between the computer system and the various valves and sensors;

FIG. 13A illustrates a view of the spacer ring;

FIG. 13B illustrates a side view of a wafer holder;

FIG. 13C illustrates a side view of the chamber insert for the drying chamber;

FIG. 13D illustrates a side view of the first embodiment of the sample holder according to the present invention;

FIG. 13E illustrates a top view of the first embodiment of the sample holder according to the present invention;

FIG. 13F illustrates a top view of the second embodiment of the sample holder, in a position to provide fluid containment, according to the present invention;

FIG. 13G illustrates a side view of the second embodiment of the sample holder, in a position to provide fluid containment, according to the present invention;

FIG. 13H illustrates a top view of the second embodiment of the sample holder, in a position to allow fluid flow, according to the present invention;

FIG. 13I illustrates a side view of the second embodiment of the sample holder, in a position to allow fluid flow, according to the present invention;

FIG. 13J illustrates a side view of the third embodiment of the sample holder according to the present invention;

FIG. 13K illustrates an enlarged side view of the plug seal of the third embodiment of the sample holder, in a position to provide fluid containment, according to the present invention;

FIG. 13L illustrates an enlarged side view of the plug seal of the third embodiment of the sample holder, in a position to allow fluid flow, according to the present invention;

FIG. 13M illustrates a side view of the fourth embodiment of the sample holder according to the present invention;

FIG. 13N illustrates a side view of the plug seal of the fourth embodiment of the sample holder, in a position to provide fluid containment, according to the present invention;

FIG. 13O illustrates a side view of the plug seal of the fourth embodiment of the sample holder, in a position to allow fluid flow, according to the present invention;

FIG. 14 illustrates the process steps executed by the computer system when controlling a critical point drying process in the drying chamber;

FIGS. 15A-15F illustrate the process steps, in more detail, executed by the computer system when controlling a critical point drying process within the drying chamber; and

FIG. 16 is a table showing the valve openings and closings for each drying mode controlled by the computer system.

#### DESCRIPTION OF THE EXEMPLARY EMBODIMENTS

[0033] Prior to describing the aspects of the present invention, some details concerning certain terms of art will be provided to facilitate the reader's understanding of the present invention and to set forth the meaning of various terms.

[0034] As used herein, the term "computer system" encompasses the widest possible meaning and includes, but is not limited to, microprocessors, standalone processors, networked processors, mainframe processors, and processors in a client/server relationship. The term "computer system" is to be understood to include at least a memory and a processor. In general, the memory will store, at one time or another, at least portions of executable program code, and the processor will execute one or more of the instructions included in that executable program code.

[0035] As used herein, the term "embedded computer system" includes, but is not limited to, an embedded central processor and memory bearing object code instructions. Examples of embedded computer systems include, but are not limited to, personal digital assistants, cellular phones and digital cameras. In general, any device or appliance that uses a central processor, no matter how primitive, to control its functions can be labeled as having an embedded computer system. The embedded central processor will execute one or more of the object code instructions that are stored on the memory. The embedded computer system can include cache memory, input/output devices and other peripherals.

[0036] It will be appreciated that the term "predetermined operations" and the term "computer system software" mean substantially the same thing for the purposes of this description. It is not necessary to the practice of this present invention that the memory and the processor be physically located in the same place. That is to say, it is foreseen that the processor and the memory might be in different physical pieces of equipment or even in geographically distinct locations.

[0037] As used herein, one of skill in the art will appreciate that "media" or "computer-readable media" may include a diskette, a tape, a compact disc, an integrated circuit, a cartridge, a remote transmission via a communications circuit, or any other similar medium useable by computers. For example, to distribute computer system software, the supplier might provide a diskette or might transmit the instructions for performing predetermined operations in some form via satellite transmission, via a direct telephone link, or via the Internet.

[0038] Although computer system software might be "written on" a diskette, "stored in" an integrated circuit, or "carried over" a communications circuit, it will be appreciated that, for the purposes of this discussion, the computer usable medium will be referred to as "bearing" the instructions for performing predetermined operations. Thus, the term "bearing" is intended to encompass the above and all equivalent ways in which instructions for performing predetermined operations are associated with a computer usable medium.

[0039] Therefore, for the sake of simplicity, the term "program product" is hereafter used to refer to a computer useable medium, as defined above, which bears instructions for performing predetermined operations in any form.

[0040] A detailed description of the preferred embodiments of the present invention will now be given referring to the accompanying drawings wherein like reference numerals refer to similar parts in the several views.

[0041] The general operation of a critical point dryer will be provided to facilitate the reader's understanding of the present invention. This description of the general operation of a critical point dryer is by no means limiting on the operation of the present invention.

[0042] The first step in a critical point drying operation is to cool the drying chamber to a temperature that will condense the transitional fluid to be added later. A cooling fluid flows around the drying chamber and evaporates in a heat exchange relationship with the drying chamber. Preferably, the cooling fluid is liquid carbon dioxide. If a closed loop refrigeration system is used to cool the drying chamber, Freon™ or its equivalent can also be used. Next, the treated specimen is placed in the drying chamber along with an amount of the dehydrating fluid, such as ethanol, methanol or acetone. The specimen has previously been dehydrated with the dehydrating fluid. After the treated specimen has been placed in the drying chamber and the cover secured, a transitional fluid fills the drying chamber. After the drying chamber is filled, the dehydrating fluid is purged from the treated specimen in the drying chamber. Preferably, the transitional fluid is liquid carbon dioxide. The purging of the

dehydrating fluid is controlled over a predetermined time period. In an aspect of the invention, the critical point drying apparatus cycles through another filling of the drying chamber with the transitional fluid to ensure that the transitional fluid completely fills the drying chamber. The drying chamber is then heated to raise the transitional fluid to its critical point pressure and critical point temperature, thereby reaching critical point equilibrium. Once critical point equilibrium is reached, the equilibrium is maintained for a certain length of time. After the equilibrium time period has expired, the drying chamber temperature is maintained while the drying chamber pressure is reduced or bled-off very slowly. When the drying chamber pressure drops below a predetermined threshold, the drying chamber is then vented to release any residual pressure that may hinder removal of the chamber cover and the chamber heat is shut off.

[0043] Referring to FIG. 1, a perspective view showing the external configuration of an embodiment of the critical point drying apparatus 1 is shown. The housing 24 encloses the internal valves, wiring, piping, switches, relays and computer system components that make up the critical point drying apparatus 1. A power switch 22 applies electrical power to the dryer through a fuse. The operation indicator lights indicate the individual operation that is being undertaken in the drying chamber 40. Preferably, the operation indicators are light emitting diodes (LED). The critical point drying apparatus 1 has the following operation indicators: cool LED 16, fill LED 17, purge LED 18, heat LED 19, bleed LED 20 and vent LED 21. The temperature gauge 23 and the pressure gauge 69 provide visible indicators of the present conditions within the

drying chamber 40. Transitional fluid and cooling fluid enter the critical point drying apparatus 1 through the inlet port 52. Exhausted cooling fluid exits the critical point drying apparatus 1 through the cool exit port 51. Purged dehydrating fluid and exhausted transitional fluid exits the critical point drying apparatus 1 through purge port 50.

[0044] Referring to FIG. 2, a perspective view showing the external configuration of another embodiment of the critical point drying apparatus 1' is shown. The critical point drying apparatus 1' is shown with a cool supply port 71. The cool supply port 71 is connected to a closed loop refrigeration system. Cooling fluid circulates through the critical point drying apparatus 1' by entering the cool supply port 71 and exiting through the cool exit port 51. Transitional fluid enters the critical point drying apparatus 1' through the inlet port 52. Purged dehydrating fluid and exhausted transitional fluid exits the critical point drying apparatus 1' through purge port 50.

[0045] Referring to FIG. 3, a top view of the drying chamber 40 is illustrated. The placement of the various inlets and outlets shown in FIG. 3 is by no means limiting and is shown for illustration purposes only. The cross-sectional view along lines I-I includes the fill inlets and the purge ports and is shown in more detail in FIG. 6. The cross-sectional view along lines II-II includes the heater and is shown in more detail in FIG. 7. The cross-sectional view along lines III-III includes the cool inlet and the cool outlet and is shown in more detail in FIG. 8. As shown in FIG. 3, the fill inlets 43,44 have an angled portion 39 that is angled relative to the drying chamber wall 47 such that when the transitional

fluid enters the drying chamber 40, it will flow into the drying chamber 40 in a swirling fashion. The swirling of the transitional fluid as it enters the drying chamber 40 allows for an even and thorough purge of the intermediary fluid from the drying chamber 40.

[0046] Referring to FIG. 4, a top view of the chamber cover 75 is illustrated. Cross-section IV-IV includes the mounting stud holes 76 and is shown in more detail in FIG. 5. The chamber cover 75 has a cover viewing port 80 for observing the interior of the drying chamber 40. Preferably, the mounting stud holes 76 are evenly arranged around the perimeter of the chamber cover 75.

[0047] Referring to FIG. 5, the cross-section of the chamber cover 75 along lines IV-IV is illustrated in more detail. This is the cross-section along lines IV-IV referred to by FIG. 4. The chamber cover 75 is secured by securing knobs (not shown) and is provided with a cover viewing window 78 for viewing the specimen during the operation of the critical point drying apparatus 1.

[0048] The cover viewing window 78 is mounted on the axial center of the chamber cover 75. Preferably, the cover viewing window 78 comprises quartz or an equivalent material. The operator can view the interior of the drying chamber 40 through the cover viewing port 80 in the chamber cover 75 and monitor the progress of the critical point drying sequence. The cover viewing window 78 is mounted with a precision machined viewing window gasket 77 that is pressure fit into the cover viewing port 80 in the chamber cover 75. Preferably, the viewing window gasket 77 comprises Teflon™ or an equivalent material. The viewing window gasket 77 holds the cover viewing window 78 in place, thereby

providing a seal that withstands high pressure and will not be damaged by the intermediary fluids.

[0049] In an aspect of the invention, the chamber cover 75 has a concave surface 79 on its underside that has a concave pitch. The concave pitch acts to evenly displace the internal pressure of the drying chamber 40 exerted on the chamber cover 75. The concave surface 79 allows the critical point drying apparatus 1 to accommodate large specimens without warping the chamber cover 75. For example, wafers used in integrated circuit manufacturing can be upwards of twelve inches in diameter. A conventional chamber cover would likely warp and be difficult to remove from the drying chamber.

[0050] Referring to FIG. 6, the cross-section of the drying chamber 40 along lines I-I will be described in more detail. The drying chamber 40 is also provided with a fill inlets 43,44 and a purge outlets 45,46 that allow the transitional fluid to fill and flow through the chamber. As the size (i.e. diameter) of the drying chamber 40 increases, the fill inlets 43,44 and the purge outlets 45,46 serve to increase the efficiency of the critical point drying apparatus 1. The fill inlet 43,44 has an angled portion 39 that is angled relative to the drying chamber wall 47 such that when the transitional fluid enters the drying chamber 40, it will flow into the drying chamber 40 in a swirling fashion. The swirling of the transitional fluid as it enters the drying chamber 40 allows for an even and thorough purge of the intermediary fluid from the drying chamber 40. A small chamber may require only a single fill inlet 43 and a single purge outlet 45 to adequately flow the transitional fluid through the drying chamber 40. However,

as drying chamber size increases, a plurality of fill inlets 43,44, are used and all have an angled portion 39 that is arranged at an angle relative to the drying chamber wall 47, which efficiently circulates the incoming transitional fluid. An added benefit of multiple fill inlets is that the drying chamber 40 fills at a much faster rate, thereby inducing less disturbance to the specimen situated in the drying chamber 40. Similarly, a large drying chamber has a plurality of purge outlets 45,46 situated at the lowest point of the drying chamber 40 that aids in the collection and purging of the intermediary fluid.

**[0051]** The drying chamber 40 has a chamber viewing window 114 mounted in the axial center of the drying chamber 40. Preferably, the chamber viewing window 114 comprises quartz or an equivalent material. The chamber viewing window 114 is lighted from below and the operator can view the interior of the drying chamber 40 through the cover viewing port 80 in the chamber cover 75 and monitor the progress of the critical point drying sequence. The chamber viewing window 114 is mounted with a precision machined viewing window gasket 115 that is pressure fit into a chamber viewing port 116 in the bottom of the drying chamber 40. Preferably, the viewing window gasket 115 comprises Teflon™ or an equivalent material. The viewing window gasket 115 holds the chamber viewing window 114 in place, thereby providing a seal that withstands high pressure and will not be damaged by the intermediary fluids.

**[0052]** Mounting studs 110 extend upward through the chamber cover 75 and in conjunction with internally threaded securing knobs (not shown), fixably and sealably mount the chamber cover 75 to the drying chamber 40. By drilling

through the drying chamber wall 47 into the mounting studs 110, stainless steel mounting pins 111 can be inserted into the mounting studs 110 to prevent any movement.

[0053] The bottom of the drying chamber 40 has a chamber concave surface 48 with a concave pitch. The concave pitch acts to each active to evenly displace the internal pressure of the drying chamber 40. The chamber concave surface 48 allows the intermediary fluid to collect and exit through the purge ports 45,46 that are located at the lowest point of the drying chamber. The concavity thus assists with dispersing the internal pressure and with the complete purging of the intermediary fluid through the bottom of the drying chamber 40. The chamber concave surface 48 allows the critical point drying apparatus 1 to accommodate large specimens without warping in the chamber cover 75.

[0054] A cover gasket 113 provides a seal between the chamber cover 75 and the top portion of the drying chamber 40. A seal groove 112 is formed in the top portion of the drying chamber 40 that directly opposes the chamber cover 75. The cover gasket 113 is set into the seal groove 112, and projects slightly above the top surface of the drying chamber 40, thereby ensuring a tight seal when the chamber cover 75 is secured. Due to the pressure in the drying chamber 40, the cover gasket 113 must be made from a material that is inert to the fluids used in the drying chamber 40 and must be able to withstand the chamber pressure without deformation. Preferably, the cover gasket 113 comprises Teflon™ or an equivalent material.

[0055] Referring to FIG. 7, the cross-section of the drying chamber 40 along lines II-II is illustrated in more detail. The drying chamber 40 also has at least one heater 32 mounted in the drying chamber wall 47 to heat the transitional fluid above the critical point. Preferably, the heater 40 is a wire-wound resistance heater that is controlled by the computer system 99.

[0056] A temperature sensor (not shown) is mounted in the wall of the drying chamber 40 is connected to a temperature gauge 73 to provide an indication of the drying chamber temperature. In addition, three other temperature sensors are mounted on the drying chamber wall 47. A heat sensor 31 (normally closed) opens when the temperature in the drying chamber 40 reaches a predetermined level and the opening of the heat sensor 31 is monitored by the computer system 99. Preferably, the heat sensor 31 is a thermostatic sensor and opens when the drying chamber temperature exceeds 42° C. A cool sensor 32 (normally closed) opens when the temperature in the drying chamber 40 drops past a predetermined level and the opening of the cool sensor 32 is monitored by the computer system 99. Preferably, the cool sensor 32 is a thermostatic sensor and opens when the drying chamber temperature is less than 5° C. Finally, the safety sensor 30 ensures that the heater 33 does not raise the drying chamber temperature past a predetermined safety level. If the drying chamber temperature exceeds the predetermined safety level, all power to the heater 33 is cut off. Preferably, the safety sensor 32 is a thermostatic sensor and opens when the drying chamber temperature exceeds 50° C.

[0057] Referring to FIG. 8, the cross-section of the drying chamber 40 along lines III-III is illustrated in more detail. A cooling fluid is circulated to cool the drying chamber wall 47 by passing into the cool inlet 41 through the walls of the drying chamber and the connecting line 49 and out through the cool outlet 42. Preferably, liquid carbon dioxide is used as a cooling fluid. The cooling fluid cools the drying chamber 40 by adiabatic cooling, which is turned on and off automatically via thermostatic controls. The fittings and tubing through which the cooling fluid flows is preferably stainless steel. In order to protect the integrity of the specimen, all external and internal surfaces of the critical point drying apparatus 1 are both chemically and moisture resistant, and all internal surfaces are inert to the intermediary and transitional fluids, such as liquid carbon dioxide and ultra-pure alcohol. In addition, all internal and external surfaces are grounded to guard against static discharge that is harmful to semiconductor wafers.

[0058] Referring to FIG. 9, a schematic diagram showing the flow of fluids through the drying chamber 40 and the various control valves is illustrated. The transitional fluid is also utilized to cool the drying chamber 40 through the cooling circuit shown. The transitional fluid, preferably liquid carbon dioxide, is provided at the inlet port 52 and then flows through a filter assembly 53 to the 3-way tee 54. The filter assembly 53 removes any particulate matter from the transitional fluid prior to entering the various valves of the critical point drying apparatus 1. Preferably, the filter assembly 53 is a stainless steel filter that removes particulate up to 0.5 microns in diameter in order to protect the specimen

in the drying chamber 40, as well as the critical point drying apparatus valves. Preferably, all tubing, fittings, valves, etc. are stainless steel. In addition, a rupture disc that will burst and evacuate the system in case of an undesirable rise in pressure protects the entire pressure system (not shown).

[0059] The transitional fluid is then piped to a pair of computer system controlled solenoid operated valves: the fill valve 56 and the cool valve 55. The fill valve 56 and cool valve 55 also comprise metering valves to control the flow rate of fluids through the valves.

[0060] When the computer system 99 energizes the cool valve 55 solenoid, the cool valve 55 supplies the transitional fluid through the metering valve to the drying chamber 40 at the cool inlet 41. The metering valve regulates the flow of transitional fluid through the wall of the drying chamber. When the cool valve 55 is energized, the transitional fluid flows out from the cool valve 55 and through the connection line to the drying chamber 40, wherein the transitional fluid is evaporated and ducted throughout the wall of the drying chamber 40. The flow is from the cool inlet 41 through the wall of the drying chamber 40 to the cool outlet 42. The warmed vaporized cooling fluid is ducted out of the critical point drying apparatus 1 at the cool port 51. If a closed loop refrigeration system is used, the cooling fluid is cycled back to a refrigeration unit. The closed loop system may use a refrigerant other than liquid carbon dioxide, such as Freon™.

[0061] When the computer system 99 energizes the fill valve 56, transitional fluid flows into the fill inlets 43,44 to fill or purge the drying chamber 40. When the drying chamber 40 is being filled with the transitional fluid, the

computer system 99 energizes the fill valve 56, and transitional fluid flows from the fill valve 56, through a check valve 57, a 4-way tee 58 and into the drying chamber 40 through the fill inlets 43,44. The in-line check valve 57 protects the fill valve from any backflow to the drying chamber.

[0062] A connection line from the 4-way tee 58 is connected to a high-pressure sensor 67, a low-pressure sensor 68 and a pressure gauge 69. The low-pressure sensor 68 opens when the chamber pressure drops below a predetermined low pressure point, and the opening of the low-pressure sensor 68 is monitored by the computer system 99. An acceptable range for the predetermined low pressure point is from 100 to 600 psi. Preferably, the predetermined low pressure point is 400 psi.

[0063] The high-pressure sensor 67 opens when the chamber pressure exceeds a predetermined high pressure point, and the opening of the high-pressure sensor 67 is monitored by the computer system 99. An acceptable range for the predetermined high pressure point is from 1175 and 1600 psi. Preferably, the predetermined high pressure point is 1200 psi.

[0064] A pressure relief valve (not shown) is also connected to the pressure sensors. The pressure relief valve will release the pressure from the critical point drying apparatus 1 when the drying chamber pressure exceeds a predetermined limit. The pressure relief valve is heated and the heating of the pressure relief valve is thermostatically controlled independent of the computer system 99. An acceptable range for the pressure relief valve is from 1200 to 1600 psi. Preferably, the pressure relief valve opens at 1250 psi.

[0065] A rupture disc 72 is also connected to the pressure sensors. The rupture disc 72 acts as an additional safety feature and is set to burst when the drying chamber pressure goes above a predetermined limit. An acceptable range for the rupture disc 72 is from 1900 to 3000 psi. Preferably, the rupture disc 72 ruptures at 2100 psi.

[0066] When the intermediary fluid is to be purged from the drying chamber 40, the computer system 99 commands the purge valve 63 to open. The purge valve 63 is a solenoid valve. The intermediary fluid is forced from the drying chamber 40 through the purge outlets 45,46 in the drying concave surface 48 and into the connection line through a filter assembly 61, which is connected to the purge valve 63. The purge valve 63 is heated to prevent the purge valve 63 from freezing when the transitional fluid or the intermediary fluid passes through the purge valve 63. The heating of the purge 63 valve is thermostatically controlled independent of the computer system 99. The purge valve 63 also includes a metering valve to control the flow rate at which the transitional fluid or intermediary fluid is purged. Attached to the exiting end of the purge metering valve is a check valve 65 to prevent fluid backflow through the purge valve 63 into the drying chamber 40. The purged fluid exits the critical point drying apparatus 1 through the purge outlet 50. Throughout the entire purging process, the computer system 99 monitors the drying chamber temperature and keeps the drying chamber 40 below a predetermined temperature, preferably 5° C or less.

[0067] The cycle time for executing a purge of the intermediary fluid from the drying chamber 40 is controlled by the computer system 99. Preferably, the

purge time is adjusted by a purge timing control 25 that is located on the housing 24 of the critical point drying apparatus 1. After the purge cycle for the purging of the intermediary fluid is complete, the computer system 99 closes the purge valve 63 and allows the fill valve 56 to continue filling the drying chamber 40 with transitional fluid. This ensures the transitional fluid fills the drying chamber 40 completely. The computer system 99 then advances the drying chamber 40 into the heating cycle.

[0068] The computer system 99 activates the heater 32 to raise the transitional fluid to its critical point pressure and critical point temperature, thereby reaching critical point equilibrium. Preferably, the heater 32 raises the drying chamber temperature to at least 31° C or greater, which, in turn, causes the temperature and pressure of the transitional fluid to reach its critical point temperature and pressure.

[0069] After the computer system 99 has determined that the specimen has been at the critical point equilibrium for a sufficient amount of time, the computer system 99 commands the bleed valve 62 to open, thereby allowing the transitional fluid to exhaust out of the drying chamber 40 and exit the critical point drying apparatus 1 through the purge outlet 50. The bleed valve 62 is a solenoid valve. When the transitional fluid is exhausted, it flows from the drying chamber 40 into the bleed valve 62 and then into the check valve 64. The check valve 64 prevents backflow from backing through the bleed valve 62 into the drying chamber 40. The bleed valve 62 also comprises a metering valve to control the bleed rate. Preferably, the metering valve allows the system pressure to decrease at a rate of

100 psi/minute. This bleed rate prevents the transitional fluid from recondensing. In addition, the bleed valve 62 is thermostatically heated to prevent the bleed valve 62 from freezing as the transitional fluid flows through it. During the bleed process, the computer system 99 maintains the drying chamber temperature at 31° C or above. This temperature level prevents recondensation on the specimen. When the drying chamber pressure is reduced to 400 psi, the computer system 99 turns off the heater 32 and switches from bleed to vent mode. The computer system 99 commands the bleed valve 62 to close and the purge valve 63 to open. This returns the drying chamber to atmospheric pressure quicker.

[0070] Referring to FIG. 10, in another aspect of the invention, the drying chamber 40 can be cooled using a closed loop refrigeration system. The closed loop system is coupled to the drying chamber 40 and allows the drying chamber 40 to be cooled without blasting liquid carbon dioxide through the drying chamber 40. This feature of the present invention provides for savings on the amount of carbon dioxide that is used during a specimen drying sequence. In addition, the closed loop system may use a refrigerant other than liquid carbon dioxide, such as Freon™. Cooling fluid enters the critical point drying apparatus 1 through cool supply port 71 and is piped to the cool valve 55. A common supply for cooling fluid and transitional fluid is not used.

[0071] Referring to FIG. 11, a condenser collector 130 allows the intermediary fluid (i.e., the dehydrating fluid) to be purged from the drying chamber 40 in a safer and more controlled manner. The condenser collector 130 is connected to the purge port 50. Preferably, methyl alcohol or ethyl alcohol is



software instructions adapted to enable the computer to control a critical point drying chamber and its associated valve and heaters. The computer system 99 includes a memory 101 that stores the software instructions adapted to enable the computer system 99 to control the drying chamber 40 and associated valves. The computer system 99 also comprises an I/O port that allows the computer system 99 to be re-programmed and to upload/download data. As shown in FIG. 12, the computer system 99 receives inputs from the temperature and pressure sensors through the data lines 103. Commands to open or close the cool valve 55, the fill valve 56, the bleed valve 62 and the purge valve 63 are sent over the data lines 103. Finally, the computer system 99 receives commands from the indicator switches on the housing 24 that indicate the various modes, and the computer system 99 also lights the appropriate indicator switch to inform the operator which mode is currently being executed.

[0074] Referring to FIGS. 13A-13G, when the critical point drying apparatus 1 will be used to process semiconductor wafers, the drying chamber must be outfitted with a mechanism to hold the semiconductor wafer in a suspended position so that the drying process can be achieved. The critical point drying apparatus 1 that are designed to process semiconductor wafers are outfitted with a wafer holder 124, a spacer ring 120 and a chamber insert 121. Preferably, these components come in different sizes to accommodate different wafer sizes, and are made out of Teflon™ or an equivalent material.

[0075] The spacer ring 120 is placed in the bottom of the appropriate wafer holder 124, then a semiconductor wafer can be placed on top, and another spacer

ring 120 can then be put in place so that another semiconductor wafer can be added. This method of suspended stacking allows multiple wafers to be successfully processed during one run.

[0076] Since semiconductor wafers come in several different sizes, there are several different size wafer holders 124. When a smaller wafer holder 124 is needed to process a semiconductor wafer, a chamber insert 121 is used to hold the wafer holder 124 in place and reduce the amount of transitional fluid used. For example, if the drying chamber 40 has a six and one half inch internal diameter and the operator wishes to process a four inch wafer, then a chamber insert 121 is put into place (having a six inch outer diameter with cavity 123 that has a four inch internal diameter). The wafer then is placed in the four-inch wafer holder 124 that is placed inside the chamber insert 121.

[0077] The wafer holder 124 is comprised of a body 125 and a handle 128. In the exemplary embodiment shown in FIG. 13B, the body 125 has a plurality of fluid holes 127 that allow the transitional fluid to reach the semiconductor wafers stacked inside the body 125.

[0078] The wafer holder 124 can be further used through many steps of fabrication of silicon wafer substrates. This process involves submerging the substrates into baths of hydrofluoric acid, rinsing them in de-ionized water (D.I.), and then placing them into baths of alcohol for dehydration prior to being placed into the chamber of the critical point dryer for further processing.

[0079] While the wafers are in each bath of fluid and during the time they are being processed by the critical point dryer, it is advantageous to have fluid

flow around them. This is accomplished by allowing holes similar to fluid holes 127 in FIG. 13B. However, when the wafers are being transferred between baths prior to and while in the critical point dryer, it is preferable, but not necessary, for the holder that transports the wafers to retain fluid inside of the holder 124 in such a way as to keep the wafers submerged in fluid at all time during the transfer between steps.

**[0080]** It is therefore preferable, but not necessary, to provide a holder that can behave with a porous nature when fluid exchange is desired and act as a holding container when transferring the wafers between processing steps.

**[0081]** Several exemplary embodiments of such holders will now be described. These embodiments are examples of holders that provide structure and function for the invention to operate, but they are not intended to limit the scope of the invention in any way only to such specific embodiments. Further, these exemplary embodiments are not intended to be limited to use in the exemplary embodiments of a critical point dryer disclosed herein.

**[0082]** An exemplary embodiment of a holder is shown in FIGS. 13D and 13E. The holder 140 includes outer section 147 and a generally cup shaped inner base section 146. Outer section 147 includes a handle 145 and an annular closing wall 141. Inner base section 146 includes annular retention wall 142, flow holes 143 and standoffs 144.

**[0083]** The holder 140 has two positions corresponding to a position to allow fluid flow, and a position to provide fluid containment. When holder 140 is in a position to provide fluid containment, outer section 147 is placed over

inner base section 146. When outer section 147 is placed over inner base section 146, the annular closing wall 141 closes off flow holes 143 in such a way as to prevent any fluid contained within inner base section 146 to escape through the holes. As can be seen in FIG. 13E, the annular closing wall 141 has only a slightly larger diameter than the annular retention wall 142, in order to provide a tight fit to provide fluid containment, and also to allow the holder 140 to be moved by an operator using handle 145.

[0084] When holder 140 is in a position to allow fluid flow, outer section 147 is removed from interaction with inner base section 146. Flow holes 143 are now not covered by annular retention wall 141, and allow fluid to flow in and out of the holes.

[0085] Another exemplary embodiment of a holder is shown in FIGS. 13F - 13I. Holder 150 includes handle 155 and holding section 151. The holding section 151 comprises a generally cup shaped inner holding section 153 with flow hole 156, and outer retaining section 152 with fluid hole 154.

[0086] The holder 150 has two positions corresponding to a position to allow fluid flow, and a position to provide fluid containment. When holder 150 is in a position to allow fluid containment, as illustrated in FIGS. 13F and 13G, outer retaining section 152 is rotated around inner holding section 153 in such a way as to not allow flow holes 154 and 156 to communicate with each other. When the flow holes 154 and 156 cannot communicate with each other, fluid that is contained within the inner holding section 153 cannot flow through the holes.

[0087] When holder 150 is in a position to allow fluid flow, as illustrated in FIGS. 13H and 13I, outer retaining section 152 is rotated around inner holding section 153 in such a way as to allow flow holes 154 and 156 to communicate with each other. When the flow holes 154 and 156 communicate with each other, fluid can flow into and out of the holes.

[0088] Another exemplary embodiment of a holder is shown in FIGS. 13J - 13L. Holder 160 includes handle 165 and generally cup shaped holding section 161. Holding section 161 includes bottom section 168, plug seal 163, and resilient member 164. Bottom section 168 has flow holes 162.

[0089] The holder 160 has two positions corresponding to a position to allow fluid flow, and a position to provide fluid containment, which are illustrated by the alternative positions of plug seals 163 in FIG. 13J. When holder 160 is in a position to allow fluid containment, pressure is not applied to the holder, which allows plug seal 163 to remain seated in flow hole 162, as illustrated in FIG. 13K. When fluid flow is desired, pressure may be applied to holder 160, which causes plug seal 163 to move vertically downward in a direction of arrows 166, as illustrated in FIG. 13L. When plug seal 163 moves downward, it unseals hole 162 and allows fluid to flow through the hole. Resilient member 164 will return the plug seal 163 to a position that seals hole 162 when pressure on the holder 160 is removed.

[0090] Another exemplary embodiment of a holder is shown in FIGS. 13M-13O. Holder 170 includes handle 175 and generally cup shaped holding section

171. Holding section 171 includes bottom section 178, plug seal 173, and plug actuating surface 174. Bottom section 178 has flow holes 172.

[0091] The holder 170 has two positions corresponding to a position to allow fluid flow, and a position to provide fluid containment, which are illustrated by the alternative positions of plug seals 173 in FIG. 13M. When holder 170 is in a position to allow fluid containment, pressure is not applied to plug actuating surface 174, which allows plug seal 173 to remain seated in flow hole 172, as illustrated in FIG 13N. When fluid flow is desired, pressure may be applied to plug actuating surface by placing the holder 170 into a position such that it is resting on an opposing surface (not shown) (i.e., a critical point dryer chamber bottom or any other surface). The opposing surface will engage the plug actuating surface 174, and the weight of holder 170 will cause the holder to settle onto the surface, which in turn will cause the plug seal 173 to move vertically upward, as illustrated in FIG. 13O. When plug seal 173 moves upward, it unseals hole 172 and allows fluid to flow through the hole 172. Plug seal 173 will return to a position that seals hole 172 when holder 170 is removed from the opposing surface and pressure is removed from plug actuating surface 174.

[0092] Referring to FIG. 14, the software instructions can be resident on the microprocessor 100 or stored on a separate memory 101. At Step S1000, the computer system 99 executes software instructions to cool the drying chamber 40 to a first chamber temperature. At S1100, the computer system 99 executes software instructions to fill the drying chamber 40 with a transitional fluid having a critical point temperature and critical point pressure while maintaining the

[illegible]

**[0093]** The software instructions adapted to enable the computer system 99 to control the drying chamber 40 and the associated valve assemblies will now be described in greater detail.

**[0094]** If commanded to by the operator, the computer system 99 executes diagnostic routines to determine if the critical point drying apparatus 1 is in working order. These diagnostics are described in greater detail below.

[0095] Next, the software instructions executed by the computer system 99 cool the drying chamber 40 to a predetermined value. As described above, at S1010, the computer system 99 commands the cool valve 55 to open, thereby

allowing the cooling fluid to flow from the inlet port 52, through the cool valve 55 and into the drying chamber walls, thereby cooling the drying chamber 40 to the desired temperature level. As described above, by commanding the cool valve 55 to open, a cooling fluid flows around the drying chamber and evaporates in a heat exchange relationship with the drying chamber 40. At S1020-1040, a determination is made whether the drying chamber 40 has reached the desired temperature. If not, the cool valve remains open.

[0096] After the specimen has been placed in the drying chamber 40 immersed in an intermediary fluid and the chamber cover 75 secured, at S1110, the computer system 99 commands the fill valve 56 to open, thereby allowing the transitional fluid to flow into the interior of the drying chamber 40. This is known as the fill mode and the computer system 99 allows the transitional fluid to flow into the drying chamber 40 for a preset amount of time. At the expiration of the preset time period, the computer system 99 commands the fill valve 56 to close. At S1115 to S1120, a determination is made if the preset time period has expired. If not, the fill valve remains open. At S1125 to S1135, an additional determination is made whether the drying chamber 40 has remained at the desired temperature. If not, the cool valve is opened to cool the drying chamber 40.

[0097] Next, at S1140, the computer system 99 executes the software instructions for purging the intermediary fluid from the drying chamber 40. This is known as the purge mode, such that the transitional fluid completely fills the drying chamber 40 and purges the intermediary fluid from the treated specimen in the drying chamber 40. As shown by S1145 to S1150, the purging of the

dehydrating fluid is controlled over a predetermined time period. As shown by S1155 to S1165, while the drying chamber 40 is being filled and purged, the computer system 99 maintains the drying chamber 40 at the predetermined temperature by controlling the cool valve 55. Preferably, the predetermined temperature is 0° C. When the computer system 99 has determined that the purging cycle has reached the end of its time period, at S1170, the fill valve 56, the purge valve 63 and the cool valve 55 are closed. The drying chamber 40 now should be completely filled by the transitional fluid.

[0098] Next, to ensure that the drying chamber 40 is completely filled, at S1210 to S1270, the computer system 99 executes software instructions that cycle back into the fill mode following the purging of the intermediary fluid. The cycling back into the fill mode ensures that the drying chamber 40 is completely filled by the transitional fluid. Therefore, the fill valve 56 is reopened by the computer system 99 to facilitate this task. During this fill, as shown by S1240 to S1260, the computer system 99 monitors the temperature and can activate the cool valve 55 if the drying chamber 40 needs to be cooled. Once complete, at S1270, all the valves are commanded to be closed.

[0099] At S1310 to S1340, after the computer system 99 has completed the second chamber fill, the drying chamber 40 is heated to pressurize the transitional fluid to its critical point as well as raise its temperature to the critical point. The computer system 99 executes software instructions to activate the heater 33 mounted in the wall of the drying chamber. The computer system 99 monitors

the temperature to ensure that the chamber temperature does not exceed a preset limit. Preferably, this temperature limit is between 48 and 50° C.

**[00100]** The software instructions also comprise instructions that command the computer system 99 to indicate to the operator that the pressure and temperature are above the critical point equilibrium. Preferably, the computer system 99 causes the heat LED 19 to flash thereby indicating that the specimens in the drying chamber 40 have reached the critical point equilibrium. The critical point equilibrium is maintained for a programmed amount of time. At S1320, if the heat thermostatic sensor 30 and the high pressure sensor 67 indicate that the critical point equilibrium has not been reached, the software instructions command the computer system 99 to indicate to the operator that there is a problem with the critical point drying sequence. At S1330, the computer system 99 will flash all the operation indication LEDs to indicate a problem with the critical point drying sequence. The operator is then allowed to press a switch to return to an earlier stage in the process at which point the computer system 99 will then take over and complete the drying sequence.

**[00101]** At S1410 to S1420, the computer system 99 executes software instructions to determine if the specimen has been at critical point equilibrium for a predetermined time period.

**[00102]** At S1510 to S1520, once the programmed amount of time at the critical point equilibrium has expired, the computer system 99 executes software instructions to bleed off the pressure in the drying chamber. The computer system 99 opens the bleed valve 62 and controls the bleeding of the pressure in

the drying chamber 40 down between 100 and 600 psi while maintaining drying chamber temperature above 31°C or above. Preferably, the low pressure threshold is 400 psi. When the computer system 99 is executing the bleed mode, the computer system 99 controls the bleeding of pressure from the drying chamber 40 in an even fashion. In dropping from 1100 psi to 400 psi, the computer system 99 allows the pressure to vent slowly from the drying chamber 40. In addition, the computer system 99 commands the heater 33 disposed on the drying chamber 40 to maintain the temperature of the drying chamber 40 at 31°C or above.

[00103] At S1610 to S1620, when the drying chamber pressure reaches 400 psi however, the software instructions command the computer system 99 to shut off the heaters and vent the remaining pressurized gas from inside the drying chamber 40 directly out the vent line. The purge valve 63 is opened and the bleed valve 62 is closed. The purge valve 63 will remain open until another run is commenced or the critical point drying apparatus 1 is powered down.

[00104] In another aspect of the present invention, the predetermined operations of the software instructions comprise instructions for conducting diagnostic testing of several sensors used to control the critical point drying sequence. The software instructions test the high pressure sensor 67 (or critical point pressure sensor), the low pressure sensor 68, the heat sensor 31 and cool sensor 32. The computer system 99 executing the software instructions indicates on the display if each of the above-identified sensors is operational. Preferably,

the computer system 99 lights an operation indication LED that indicates to the operator that the sensor is working properly.

[00105] The predetermined operations of the software instructions also comprise diagnostic instructions that support the testing of switches, their corresponding operation indication LED switches and microprocessor interrupt service routine. For example, to test the purge LED switch 18, pressing the purge LED switch 18 while the critical point drying apparatus 1 is powered up will cause the purge LED switch 18 to light if the computer system 99 is executing the software correctly. In another example, pressing the heat LED switch 19 when powering up the apparatus causes the operation indication LEDs to individually light in sequence, thereby indicating that the microprocessor clock is operating correctly. Preferably, the operation indication LEDs light sequentially in fifteen-second intervals.

[00106] The predetermined operations of the software instructions also comprise calibration instructions for the cool sensor 32. The computer system 99 will cool the drying chamber 40 independently of the cool sensor 32. The fill LED switch 17 will indicate the status of the cool sensor 32 and will allow the operator to determine if the cool sensor 32 is operating correctly or that it needs adjustment.

[00107] The predetermined operations of the software instructions also comprise instructions to ensure that the buildup of static electricity does not affect the operation indication LED switches. The computer system 99 constantly checks the status of the operation indication LED switches to ensure that they are

operating properly. If an operation indication LED has prematurely switched off, the computer system 99 determines at what point of the critical point drying sequence the critical point drying apparatus 1 is in, and then switches on the appropriate operation indication LED.

[00108] Another aspect of the present invention will now be discussed. The present invention may be embodied on a computer program product for enabling a computer system to perform critical point drying techniques when coupled to a critical point drying apparatus. The software instructions that enable the computer system to perform predetermined operations as required by the present invention are borne on a computer readable medium. The predetermined operations borne on the computer program product comprise software instructions for cooling the drying chamber to a first chamber temperature. Preferably, the first chamber temperature is between 5 and -10° C.

[00109] The predetermined operations borne on the computer program product further comprise software instructions for filling the drying chamber 40 with a transitional fluid having a critical point temperature and critical point pressure while maintaining the drying chamber 40 at the first chamber temperature such that the transitional fluid completely displaces the intermediary fluid within a first time period. As noted above, the first chamber temperature is preferably between 5 and -10° C. The predetermined operations borne on the computer program product maintains the drying chamber 40 at the first chamber temperature while the intermediary fluid is exhausted from the interior of the drying chamber 40. When the intermediary fluid is purged, the predetermined

operations borne on the computer program product ensure that the drying chamber is completely filled with transitional fluid to ensure a successful drying cycle. This is done by going through a quick fill cycle.

[00110] The predetermined operations borne on the computer program product further comprise software instructions for activating at least one heater to raise the transitional fluid to its critical point pressure and critical point temperature, thereby reaching a critical point equilibrium. Preferably, the predetermined operations borne on the computer program product command a heater 32 on the drying chamber 40 to heat the transitional fluid to at least 31° C or above. The predetermined operations borne on the computer program product also notify the operator if the critical point equilibrium was successfully reached.

[00111] The predetermined operations borne on the computer program product further comprise software instructions for maintaining the transitional fluid at the critical point equilibrium for a second time period. The predetermined operations borne on the computer program product notify the operator that the transitional fluid in the drying chamber is at its critical point equilibrium.

[00112] The predetermined operations borne on the computer program product further comprise software instructions for bleeding the transitional fluid from the drying chamber 40 while maintaining the drying chamber 40 at the second chamber temperature. Preferably, the predetermined operations borne on the computer program product command the at least one heater 32 to maintain a

drying chamber temperature of at least 31° C while the transitional fluid is bled from the drying chamber 40.

[00113] Another aspect of the present invention will now be discussed. The present invention may be embodied on an article of manufacture, which comprises a computer readable medium having stored therein a computer program to control a drying chamber during a critical point drying process. The article of manufacture comprises a computer program product that bears a first computer code segment which, when executed on a computer, cools the drying chamber 40 to a first chamber temperature. Preferably, the first chamber temperature is between 5 and -10° C.

[00114] The article of manufacture further comprises a second computer code segment which, when executed on a computer, fills the drying chamber 40 with a transitional fluid having a critical point temperature and critical point pressure while maintaining the drying chamber 40 at the first chamber temperature such that the transitional fluid completely displaces the intermediary fluid within a first time period. As noted above, the first chamber temperature is preferably between 5 and -10° C. The second computer code segment, when executed on a computer, maintains the drying chamber 40 at the first chamber temperature while the intermediary fluid is exhausted from the interior of the drying chamber. When the intermediary fluid is purged, the second computer code segment, when executed on a computer, ensures that the drying chamber 40 is completely filled with transitional fluid to ensure a successful drying cycle.



form disclosed, and modifications and variations are possible in light of the above teachings or may be acquired from practice of the present invention. The embodiments were chosen and described in order to explain the principles of the present invention and its practical application to enable one skilled in the art to utilize the present invention in various embodiments and with various modifications as are suited to the particular use contemplated.

[00119] Thus, while only certain embodiments of the present invention have been specifically described herein, it will be apparent that numerous modifications may be made thereto without departing from the spirit and scope of the present invention. Further, acronyms are used merely to enhance the readability of the specification and claims. It should be noted that these acronyms are not intended to lessen the generality of the terms used and they should not be construed to restrict the scope of the claims to the embodiments described therein.